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Indian Standard
METHODS OF CHEMICAL ANALYSIS OF FERROSILICON
PART VI DETERMINATION OF CALCIUM
(First Rivision)

First Reprint JANUARY 1998

UDC 669.15'782-198 : 543 : 546.41

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 2 ***March* 1982**

Indian Standard

METHODS OF CHEMICAL ANALYSIS OF FERROSILICON

PART VI DETERMINATION OF CALCIUM

(First Revision)

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Indian Standard

METHODS OF
CHEMICAL ANALYSIS OF FERROSILICON

PART VI DETERMINATION OF CALCIUM

(First Revision)

0. FOREWORD

0.1 The Indian Standard (Part VI) (First Revision) was adopted by the Indian Standards Institution on 29 January 1982, after the draft finalized by the Methods of Chemical Analysis Sectional Committee had been approved by the Structural and Metals Division Council.

0.2 IS : 1559-1961* which covers chemical analysis of different ferro-alloys, namely, ferrosilicon, ferrochromium, ferromanganese and speigeleisen, silicomanganese, ferromolybdenum, ferrophosphorus, ferrotitanium, ferrovanadium and ferrotungsten, was first published in 1961. While reviewing this standard, the Sectional Committee felt that the standard should be revised to cover ferrosilicon, and methods of analysis of other ferro-alloys should be covered in separate standards. During preparation of the revision of IS : 1559-1961*, it was felt convenient to cover methods of chemical analysis of ferrosilicon in different parts of the standard which on publication would supersede Section 1 Ferrosilicon of IS : 1559-1961*. The committee also decided to rewrite the methods taking into consideration the latest composition specified in IS : 1110-1981†. Though calcium and manganese are not specified in IS : 1110-1981†, the determination of these elements is covered in this standard because these are generally required from the view point of determining interference of analysis in methods for determining other elements in ferrosilicon due to presence of these elements. Moreover, the determination of these elements may be required for comprehensive analysis of ferrosilicon. This part covers the determination of calcium. The other parts cover as follows:

Part I	Determination of silicon
Part II	Determination of carbon

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*Methods of chemical analysis of ferro-alloys.

†Specification for ferrosilicon (*third revision*).

Part III Determination of sulphur
Part IV Determination of phosphorus
Part V Determination of aluminium
Part VII Determination of manganese

0.3 In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

1. SCOPE

1.1 This standard (Part VI) covers the determination of calcium (up to 3 percent *Max*) in ferrosilicon.

2. SAMPLING

2.1 It shall be drawn and prepared in accordance with IS : 1472-1977†.

3. QUALITY OF REAGENTS

3.1 Unless otherwise specified pure chemicals and distilled water (*see* IS : 1070-1977‡) shall be employed in the tests.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

4. OUTLINE OF THE METHOD

4.1 The sample is dissolved in hydrofluoric and nitric acids in a platinum crucible and the excess of acids is removed by evaporation to dryness and ignition. The residue is then fused by a mixture of sodium carbonate and potassium carbonate and the melt extracted in dilute hydrochloric acid. The solution is evaporated to syrupy consistency followed by fuming with perchloric acid. From the solution, R_2O_3 is separated by precipitation with ammonium hydroxide. From the filtrate, calcium is determined by complexometric titration with EDTA using either Calcein or Murexide as indicator.

5. REAGENTS

5.1 Hydrofluoric Acid — 40 percent.

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*Rules for rounding off numerical values (*revised*).

†Methods of sampling ferro-alloys for determination of chemical composition (*first revision*).

‡Specification for water for general laboratory use (*second revision*).

5.2 Concentrated Nitric Acid — (rd = 1·42) (conforming to IS : 264-1976*)

5.3 Perchloric Acid — 60 percent (*v/v*).

5.4 Sodium Carbonate — Solid.

5.5 Potassium Carbonate — Solid.

5.6 Dilute Hydrochloric Acid — 40 percent (*v/v*).

5.7 Ammonium Chloride — Solid.

5.8 Ammonium Hydroxide Solution — rd = 0·90 and 50 percent (*v/v*).

5.9 Sodium Hydroxide Solution — 40 percent (*m/v*).

5.10 Standard EDTA Solution (0·005 M) — Dissolve 1·864 g of the disodium salt of EDTA [$(OOC.CH_2)_2NH.CH_2 CH_2.NH (CH_2 COO)_2 Na_2.2H_2O$] in water and transfer to a 1 000-ml volumetric flask, dilute to the mark and mix.

5.11 Calcein or Murexide Indicator Solution

6. PROCEDURE

6.1 Weigh around 1 g of the dried sample in a 50-ml tall form platinum crucible. Add about 20 ml of hydrofluoric acid and cautiously add concentrated nitric acid dropwise until the vigorous reaction has subsided. Add a few drops of perchloric acid and bring to fumes on a hot-plate. Evaporate the solution to dryness on a water-bath.

6.2 Fuse the residue in the platinum crucible with about 5 g of a mixture of sodium carbonate and potassium carbonate (3:2) and extract the melt in about 50 ml of dilute hydrchloric acid. Evaporate the solution to syrupy consistency and take to fumes with about 20 ml of perchloric acid. Cool, dilute, boil, filter and wash with hot water collecting filtrate along with washings in a 500-ml beaker. Add about 3 to 4 g of ammonium chloride. Heat to boiling and precipitate mixed oxides by adding ammonium hydroxide solution (50 percent) to a slight excess. Boil, filter and wash with hot water collecting the filtrate and washings in a beaker.

6.3 Dissolve the R_2O_3 precipitate on the filter by pouring on to the filter hot dilute hydrochloric acid, wash with hot water and reprecipitate R_2O_3 and filter, collecting the filtrate and washings in the same filtrate preserved above.

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*Specification for nitric acid (*second revision*).

6.4 Evaporate the combined filtrates, if necessary, to a volume of about 200 ml. Transfer the solution to a measuring flask of 250 ml, dilute to the mark and take the aliquot. To the aliquot, add 3 ml of sodium hydroxide solution and dilute to about 100 ml. Titrate with standard solution of EDTA using Calcein or Murexide as an indicator.

6.5 Calculation

$$\text{Calcium, percent} = \frac{A \times B \times 100}{C}$$

where

A = volume in ml of standard EDTA solution required to titrate the aliquot,

B = Equivalent calcium in g per ml of standard EDTA solution, and

C = Mass in g of the sample represented by the aliquot taken.

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